Standard Operating Procedure for the 'Modified' Digestion Preparation of Water for Total Recoverable Metal Parameters

1.0 Location

This procedure is performed in the spectroscopy laboratory, room 305.

2.0 Purpose

This method is an acid digestion procedure used to prepare water and wastewater samples for analysis by furnace atomic absorption spectroscopy (GFAA), inductively coupled plasma spectroscopy (ICP), or inductively coupled plasma-mass spectrometry (ICP-MS).

3.0 Scope

This procedure prepares the analytes for analysis by GFAA, ICP, and ICP-MS and it does not include total metal parameters or marine and estuarine water samples.

4.0 Reference

Methods for the Determination of Metals in Environmental Samples, Supplement I. US EPA. May 1994. Method 200.7, revision 4.4, Method 200.8 revision 5.4 and Method 200.9, revision 2.2. Also of applicable reference is the Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WEF, 20th Ed. Method 3030.

5.0 Sample Handling and Preservation

Samples should be collected following the procedures for metals listed in the EPA document, Methods for Chemical Analysis of Water and Wastes, Rev. 3-83, Table 1.

6.0 Apparatus and Materials

6.1 50-60 mL screw top plastic cups available from various suppliers to fit Environmental Express Hot Block digestion apparatus with 10 and 50 mL graduation demarcation.

6.2 Environmental Express Hot Block 36 position digestion apparatus.

6.3 Nitric Acid -- Instra-analyzed

6.4 ASTM Type I reagent water

6.5 Digestion spike solution with the following concentration of parameters made from ICP stock standards: 2000 mg/L ea. of Na, Mg, K, Ca; 50 ppm ea. of Fe, Mn; 10 ppm ea. of Be, Al, V, Cr, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Sb, Ba, Tl, Pb, Th, U.

7.0 Procedures

7.1 Preheat the hot block to 112-115 degrees C. This was experimentally determined to reach a digestion vessel/sample temperature of approximately 95 degrees C.

7.2 Take a 50 mL aliquot from a well mixed, acid preserved sample and transfer it to a 50 mL digestion vessel. Add 2.5 mL of conc. HNO_3. Heat the sample in the hot block until the volume has been reduced to approximately 10 mL.

7.3 Allow to cool and bring back to 50 mL volume in same vessel used for digestion with ASTM Type I water and mix. Centrifuge or filter sample to remove particulates from suspension.

7.4 Prepare blanks, spiked blanks, and sample spikes as in 7.2 and 7.3. To the sample spikes, add 0.25 mL of digestion spike solution.

7.5 Record the log numbers of the digested samples on the Digestion Data Sheet. Record the log numbers of the sample spikes on the Digestion Data Sheet.

8.0 Quality Assurance/Quality Control

8.1 For each batch of samples processed, a laboratory reagent blank and a laboratory fortified (spiked) blank should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated and also in determining recovery problems not due to matrix interference.

8.2 Sample spikes should be included at a 10% frequency with at least one per batch of samples.

9.0 Documentation

Keep records of samples digested in digestion logbook which indicates sample log number, date digested and drawer of original sample storage. Also record log numbers of all spiked and duplicated samples.

10.0 Records

All recorded information shall be maintained and kept in the digestion log book.